AFRL-RX-WP-TP-2012-0369

LOW TEMPERATURE SUPERPLASTICITY OF Ti-6A1-4V PROCESSED BY WARM MULTIDIRECTIONAL FORGING (PREPRINT)

S.L. Semiatin
Metals Branch
Structural Materials Division

G.A. Salishchev, E.A. Kudrjavtsev, and S.V. Zherebtsov
Belgorod State University

July 2012
Interim

AIR FORCE RESEARCH LABORATORY
MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
July 2012	Technical Paper	1 June 2012 – 1 July 2012

4. TITLE AND SUBTITLE

LOW TEMPERATURE SUPERPLASTICITY OF Ti-6A1-4V PROCESSED BY WARM MULTIDIRECTIONAL FORGING (PREPRINT)

5a. CONTRACT NUMBER
In-house

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
62102F

6. AUTHOR(S)

S.L. Semiatin (RXCM)
G.A. Salishchev, E.A. Kudrjavtsev, and S.V. Zherebtsov (Belgorod State University)

5d. PROJECT NUMBER
4347

5e. TASK NUMBER
20

5f. WORK UNIT NUMBER
25100102

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Metals Branch
Structural Materials Division
Air Force Research Laboratory, Materials and Manufacturing Directorate
Wright-Patterson Air Force Base, OH 45433-7750
Air Force Materiel Command, United States Air Force

8. PERFORMING ORGANIZATION REPORT NUMBER
AFRL-RX-WP-TP-2012-0369

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory
Materials and Manufacturing Directorate
Wright-Patterson Air Force Base, OH 45433-7750
Air Force Materiel Command
United States Air Force

10. SPONSORING/MONITORING AGENCY ACRONYM(S)
AFRL/RXCM

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)
AFRL-RX-WP-TP-2012-0369

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution unlimited. Preprint to be submitted to Materials Science Forum.

13. SUPPLEMENTARY NOTES

The U.S. Government is joint author of this work and has the right to use, modify, reproduce, release, perform, display, or disclose the work. PA Case Number and clearance date: 88ABW-2012-2163, 12 April 2012. This document contains color.

14. ABSTRACT

Multidirectional forging has been developed to produce an ultrafine-grain (UFG) microstructure in the two-phase titanium alloy Ti-6Al-4V. A microstructure with a grain size of 135 nm was attained, enabling low-temperature superplasticity (LTSP) at 550 degrees Celsius. A total elongation of 1000% and strain-rate-sensitivity coefficient m=0.47 were obtained at the optimal strain rate of 2x10^-4 s^-1. Important features of the microstructure and superplastic behavior of the alloy are summarized in the present work. It is shown that microstructure evolution during low-temperature deformation plays a key role in superplastic flow behavior.

15. SUBJECT TERMS

low-temperature superplasticity, multidirectional forging, ultrafine grain structure, microstructure evolution

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT:	NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON (Monitor)
a. REPORT	b. ABSTRACT	c. THIS PAGE			Donna Ballard
Unclassified	Unclassified	Unclassified	SAR	8	19b. TELEPHONE NUMBER *(Include Area Code)* N/A

Low Temperature Superplasticity of Ti-6Al-4V Processed by Warm Multidirectional Forging

G.A. Salishchev [1,a], E.A. Kudrjavtsev[1,b], S.V. Zherebtsov[1,c], S.L. Semiatin[2,d]

[1] Belgorod State University, Pobeda 85, Belgorod 308015, Russia

[2] Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/RXLM, Wright-Patterson Air Force Base, OH 45433-7817 USA

[a]Salishchev@bsu.edu.ru, [b]Kudryavtsev@bsu.edu.ru, [c]ser_z@mail.ru, [d]Lee.Semiatin@wpafb.af.mil

Keywords: Low-temperature superplasticity, Multidirectional forging, Ultrafine grain structure, Microstructure evolution

Abstract. Multidirectional forging has been developed to produce an ultrafine-grain (UFG) microstructure in the two-phase titanium alloy Ti-6Al-4V. A microstructure with a grain size of 135 nm was attained, enabling low-temperature superplasticity (LTSP) at 550°C. A total elongation of 1000% and strain-rate-sensitivity coefficient m=0.47 were obtained at the optimal strain rate of 2×10^{-4} s^{-1}. Important features of the microstructure and superplastic behavior of the alloy are summarized in the present work. It is shown that microstructure evolution during low-temperature deformation plays a key role in superplastic flow behavior.

Introduction

Manufacturing methods involving severe plastic deformation and the subsequent mechanical properties of two-phase titanium alloys with an UFG microstructure have been studied extensively during the last two decades [1-5]. Different processes, including rolling, multidirectional forging (MF), equal-channel-angular pressing (ECAP), and high-pressure torsion at low temperatures have been applied to produce UFG microstructures. Recent work has shown that a UFG microstructure with a grain size of 100-150 nm can be produced by MF in the α/β titanium alloy Ti-6Al-4V [3].

The great interest in microstructure refinement is associated with significantly reduced superplastic (SP) forming temperatures by as much as a few hundred degrees [6]. For example, in Ti-6Al-4V with a grain size of 300 nm produced by MF, SP behavior was observed at 600°C and a strain rate of $5\times10^{-4}s^{-1}$; the total elongation δ and strain-rate-sensitivity coefficient m were 500% and 0.34, respectively [7]. The same alloy with the same grain size produced by ECAP showed δ = 296% and m = 0.34 at 600°C and $1\times10^{-4}s^{-1}$ [4]. An increase in the deformation temperature resulted in an enhancement of SP properties. Sheets of Ti-6Al-4V with grain size of 300-400 nm produced by MF and subsequent pack rolling showed $\delta\approx$800% at 650°C and δ>1000% at 750°C for a strain rate of $7\times10^{-4}s^{-1}$ [2].

A decrease in SP properties at lower temperatures may be expected due to both the natural retardation of diffusion-controlled processes and the increase in the fraction of the α phase in which diffusion is much slower than that in the β phase [8]. Another factor which may contribute to the decreased δ is the propensity for grain growth when the fraction of β phase decreases noticeably.

Despite extensive studies of constitutive behavior during LTSP, detailed microstructure evolution data are known only for 775 and 815°C [9]. The aim of the present work, therefore, was to quantify microstructure evolution and mechanical behavior of Ti-6Al-4V with grain size of 135 nm produced by MF during SP deformation at the extremely low temperature of 550°C.

Materials and Procedures

The program material consisted of the α/β titanium alloy Ti-6Al-4V with a nominal composition (in weight pct.) of 6.3 Al, 4.1 V, 0.18 Fe, 0.03 Si, 0.02 Zr, 0.01 C, 0.18 O, 0.01 N. It was supplied

in the form of a hot-rolled 40-mm diameter bar with a β-transus temperature of 990°C.

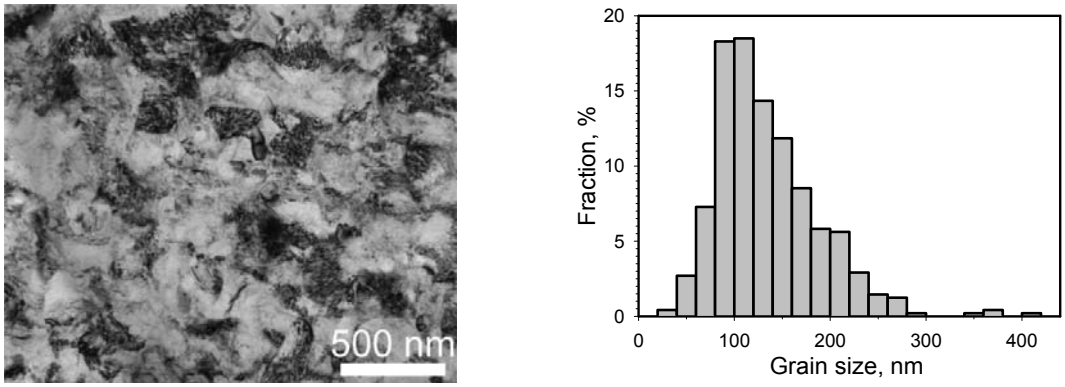
Bulk specimens measuring ∅40×60 mm with a UFG microstructure were produced initially by MF under isothermal conditions [3]. For this purpose, preforms water quenched following beta annealing were worked in the temperature interval of 700-550°C using a hydraulic press equipped with isothermal-forging tooling. The nominal strain rate was ~10^{-3} s^{-1}. Following MF, 4-mm-thick rolling performs were sectioned along the longitudinal axis by electric-discharge machining. To increase microstructure homogeneity and to form a rolling texture before SP tension testing, each plate was rolled to sheet at 475°C under isothermal conditions. The reduction per pass was 5-10%. The total reduction of was ~50%, thus yielding a final sheet thickness of 2 mm.

Tension specimens with a gage section measuring 12×3×1.5 mm were machined from the sheets. Tension tests were performed in an Instron mechanical-testing machine at temperatures between 450 and 600°C with a crosshead speed of 0.05-100 mm/min. The strain rate sensitivity *m* was evaluated using either the slope of $\log\sigma$-$\log\dot{\varepsilon}$ curves or strain-rate-change tests [6]. The apparent activation energy of plastic deformation *Q* was calculated from the semi-log dependence of the ultimate tensile strength $\ln(\sigma/G)$ on the inverse absolute temperature ($1/T$) for $\dot{\varepsilon}=5\times10^{-4}$ s^{-1} and T = 450-700°C.

X-ray investigation was carried out using a DRON-3 diffractometer with Cu-Kα radiation. The details of structural evolution were determined using a JEOL JEM-2100FX transmission electron microscope (TEM) and a Quanta 600 field-emission-gun scanning-electron microscope (SEM).

Results and Discussion

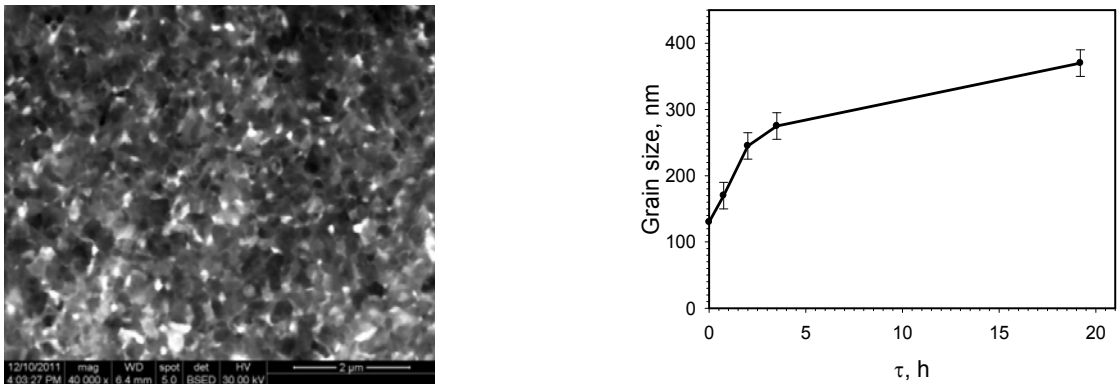
Microstructure Evolution during Annealing. After MF and rolling (referred to as the initial condition), the microstructure consisted of globular α and β grains with a mean size of 135 nm (Fig. 1a). Due to microscopic residual stresses, some fragments did not have clear boundaries. A histogram of the grain-size distribution (Fig. 1b) suggested quite good homogeneity of the microstructure. However, a small fraction of grains with a size of 300-400 nm was present.



a b

Figure 1 – Microstructure of Ti-6Al-4V in the initial condition (after MF and rolling): (a) TEM bright-field image and (b) grain-size distribution.

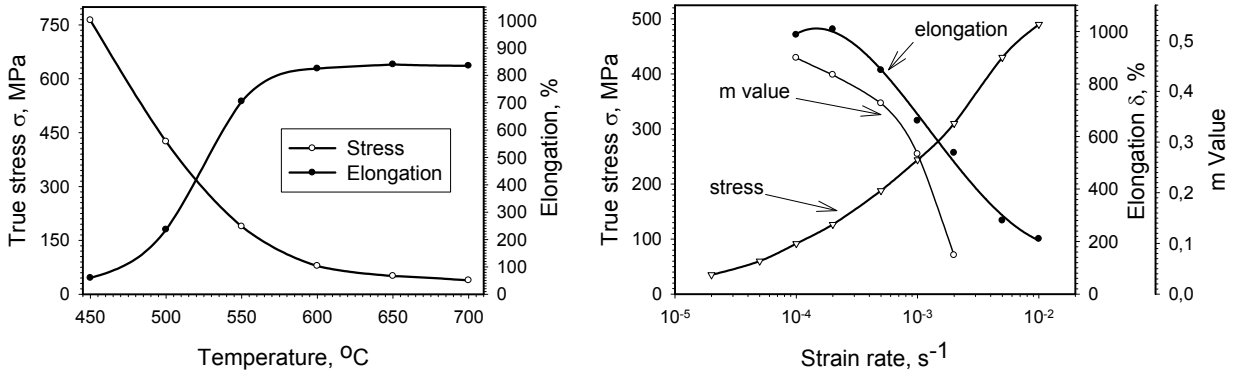
Heating to T=550°C resulted in noticeable grain growth; the kinetics of this process depended considerably on soak time (Fig. 2). In the interval of 0 - 4 hours, the grain size increased rapidly; during further soaking at 550°C, the rate of grain growth decreased. After 20 hours, the mean grain size had increased by approximately a factor of three compared to the initial condition (Fig. 2b). A backscattered electron (BSE) image revealed the distribution of the β phase in the microstructure (Fig. 2a). After a short soak time, the β phase appeared in the form of separate particles or thin laths situated between the α particles (Fig. 2a). An increase in heat treatment time to 20 hours led to coarsening and redistribution of the β particles mainly to triple points. The volume fraction of the β phase (determined by the linear intercept method) was ~10% in the initial condition and did not change noticeably during annealing.



a b

Figure 2 - (a) BSE image of Ti-6Al-4V after annealing at T=550°C for 0.5 hour and (b) grain size as a function of soak time at 550°C.

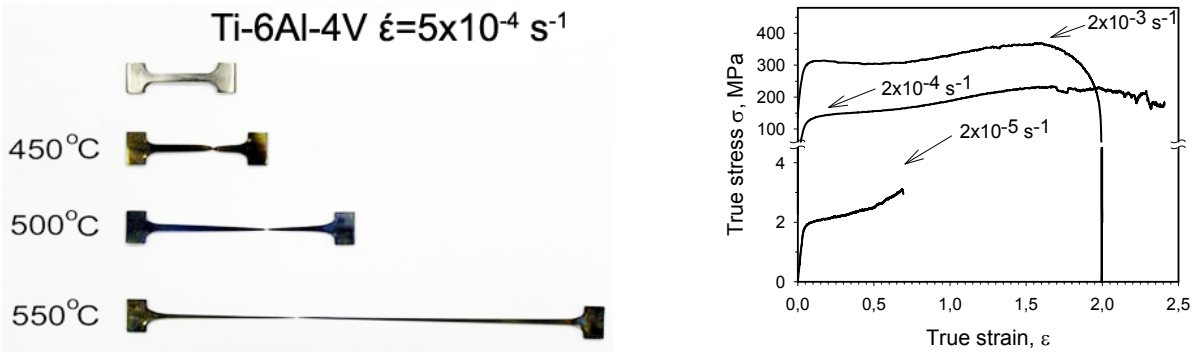
Superplastic Behavior. Figure 3 shows the mechanical properties of the UFG alloy as a function of temperature and strain rate. An increase in deformation temperature increased the ductility of the alloy and resulted in a marked decrease in flow stress (Fig. 3a). At T=550°C and $\dot{\varepsilon}=5\times10^{-4}$ s^{-1}, the total elongation δ was 640%. A further increase in temperature led to a small increase in δ, a trend likely mitigated by the oxidation effect and the formation of alpha case. An examination of specimens strained at different temperatures (Fig. 4a) showed that deformation at 550°C occurred homogeneously with obvious signs of SP flow. The effect of strain rate on the mechanical behavior at 550°C was also typical of that for SP deformation (Fig. 3b). The optimal SP parameters for the UFG alloy were δ=1000% and m = 0.47 at a strain rate of 2×10^{-4} s^{-1}.



a b

Figure 3 – Mechanical properties of the UFG alloy as a function of (a) deformation temperature for $\dot{\varepsilon}=5\ 10^{-4}$ $s^{-1}$and (b) strain rate for T=550°C. The stress level was defined at ε ~ 0.2.

The shape of the flow curves for the UFG material at T=550°C (corrected to a constant true strain rate) depended noticeably on strain rate. At 2×10^{-3} s^{-1}, the flow curve exhibited a peak flow stress at the initial stages of deformation and then near steady-state flow until fracture. A decrease in the strain rate to the optimal value of 2×10^{-4} s^{-1} changed the mechanical behavior to a marked flow-hardening response suggestive of dynamic coarsening [9]. A similar behavior was observed at the yet lower strain rate of 2×10^{-5} s^{-1}. In general, such behaviors are typical of SP flow. However, there are large differences in the levels of flow stress. For the optimal condition at 2×10^{-4} s^{-1}, the flow stress was ~130 MPa. This is an order of magnitude higher than that typically observed for high-temperature superplasticity at the same strain rate [6]. The higher flow stresses can be ascribed largely to the marked decrease in the diffusivity at the lower temperatures utilized.

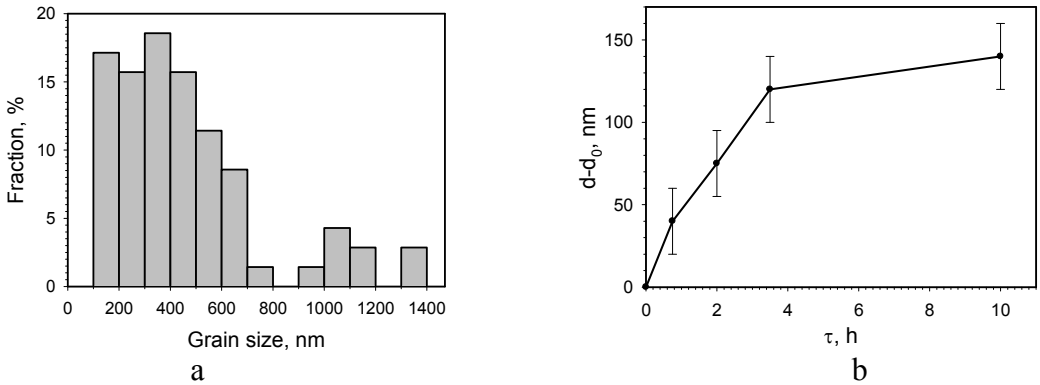


a b

Figure 4 – (a) Macrographs of test specimens of UFG Ti-6Al-4V after SP deformation at different temperatures and a strain rate of 5×10^{-4} s^{-1} and (b) true stress- true strain curves for UFG Ti-6Al-4V obtained at T=550°C and various strain rates.

Apparent Activation Energy of Plastic Deformation. The activation energy for a specific deformation process can provide insight into the rate-controlling mechanism. Under the optimal parameters of superplasticity, Q=149 kJ/mol at n=2 was obtained. In earlier works [4, 10] for the same alloy, a similar values of 176 kJ/mol for region II of superplasticity at T=600°C and 160 kJ/mol at temperatures between 650 and 955°C were reported.

Microstructure Evolution. Microstructure evolution of the UFG alloy at T=550°C as a function of strain and strain rate was quantified. Strain rate can greatly influence microstructure evolution; a decrease in strain rate often results in grain growth. For a strain ε = 100%, the mean grain size was found to be 290 nm at $\dot{\varepsilon}=5\times10^{-3}$ s^{-1}, 490 nm at $\dot{\varepsilon}=2\times10^{-4}$ s^{-1}, and 530 nm at $\dot{\varepsilon}=2\times10^{-5}$ s^{-1}. An increase in strain using the optimal strain rate for superplasticity also resulted in considerable grain growth such that a measurable fraction of grains with a size of the order of 1400 nm was observed (Fig. 5a). The plot in Fig 5b further quantifies strain-induced grain growth at 2×10^{-4} s^{-1} and T=550°C as compared to that during annealing at 550°C for equivalent times.



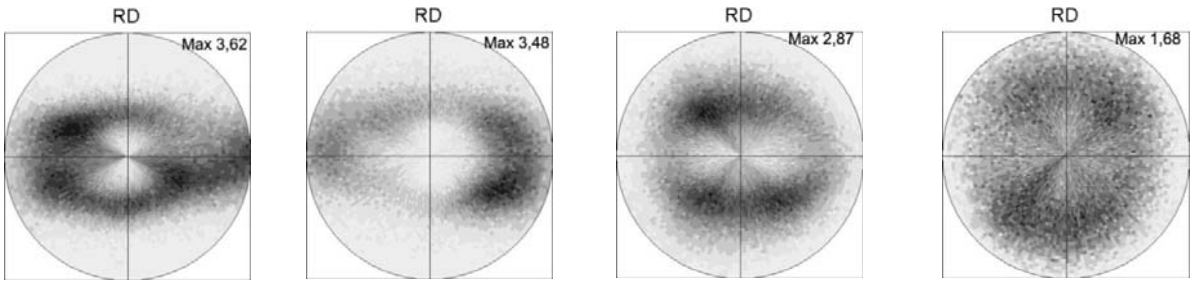
a b

Figure 5 – (a) Grain-size distribution after ε = 500% and (b) strain-induced grain growth at 2×10^{-4} s^{-1} and T=550°C in comparison with that during annealing for the same time as that needed for a given strain. In (b) d denotes the mean grain size after a given strain and d_0 is the mean grain size after annealing for the equivalent time.

Under optimal SP conditions, the strain rate sensitivity was determined to be m=0.52, 0.62 and 0.65 for elongations of 100, 200, and 500%, respectively. Consequently, despite considerable grain growth after large plastic strain, SP flow was still observed.

TEM investigation of substructure evolution during SP deformation of the UFG alloy to an elongation of 100% at T=550°C and various strain rates indicated the presence of dislocations in the α phase even after slowest strain rate of 2×10^{-5} s^{-1}. The dislocation density tended to decrease with a decrease in strain rate. However, the absolute change in comparison to the initial condition was small. The absence of porosity in the specimens deformed to fracture (ε=1000%) at 2×10^{-4} s^{-1} was aslo noted.

Texture Evolution. The evolution of the crystallographic texture during annealing and SP deformation was quite typical [6]. In the initial condition (Fig. 6a), the (0002) alpha-phase pole figure consisted of a sharp split texture with basal poles tilted ~30° from the ND toward the TD and slightly split toward the RD direction. Annealing changed neither the intensity nor the location of the poles considerably (Fig. 6b). During SP deformation, the basal poles moved toward the RD direction (Fig. 6c). The intensity of poles decreased slightly with decreasing SP strain rate. An increase in strain from 100 to 500% under the optimal strain rate of $\dot{\varepsilon}=2\times10^{-4}$ s^{-1} resulted in the formation of a diffuse pole figure with much lower intensity (Fig. 6d). The Kearn's factor calculated from the pole density [11] for the normal plane of tensile specimens decreased from 0.55 in the initial condition to 0.42 after 500% elongation at $2\times10^{-4}s^{-1}$.



a b c d

Figure 6 – $(0002)_\alpha$ pole figures taken from the normal plane of the plate produced by MF and rolling: (a) In the initial condition and (b) after annealing at 550°C for 0.5 h. $(0002)_\alpha$ pole figures measured on tension-specimen gage sections (on the surface corresponding to the normal plane of the initial sheet) after testing at $\dot{\varepsilon}=2\times10^{-4}$ s^{-1} to an elongation of (c) 100% or (d) 500%.

Discussion

UFG Ti-6Al-4V exhibited SP behavior at the extremely low temperature of 550°C. This temperature falls into the interval typically used to age the alloy (i.e., 480-595°C [12]), thus implying reasonable diffusivity. However, a decrease in temperature to 450°C (below the lower bound of this interval) resulted in an increase in flow stress to 760 MPa, a value more than 4 times that at 550°C (170 MPa) (Fig. 3a). The decrease in grain size to 135 nm made possible SP deformation with high values of strain rate sensitivity, uniform and total elongation, and flow hardening as a result of dynamic grain growth/alpha-particle coarsening under the optimal conditions. However, some features of the SP behavior are noteworthy.

It is usually thought that SP behavior in Ti-6Al-4V can be expected when the ratio of the α/β volume fractions is close to 1 [6, 12]. This is because (i) grain boundary sliding occurs more readily along interphase boundaries and (ii) microstructure stability is enhanced. A reduction of temperature to 550°C leads to a marked decrease in the fraction of the β phase to ~10%, thereby decreasing the length of the interphase boundaries and increasing microstructural instability. Indeed, the kinetics of strain-induced grain growth are much faster than those during static annealing (Fig. 5b). In addition, the fact that the tested specimens did not exhibit porosity near the fracture surface at low temperatures appears very unusual, and may be partially rationalized on the basis of grain-coarsening enhanced relaxation of intergranular stresses during SP deformation.

The mechanism of LTSP in UFG Ti-6Al-4V can be deduced based on the value of the apparent activation energy. Although the value obtained here (149 kJ/mol) is slightly lower than that reported for higher-temperature SP (176 and 160 kJ/mol [4, 10]), the difference is not large. Also, the value of activation energy obtained in the present work is similar to that reported in [13] for grain-boundary diffusion in the SP regime from 800 to 950°C for conventional Ti-6Al-4V (189 kJ/mole). Thus, it may be hypothesized that the deformation mechanism for UFG Ti-6Al-4V at lower temperatures is grain-boundary sliding accommodated by grain-boundary diffusion.

The reduction in texture intensity during SP deformation can also be ascribed to grain-boundary sliding and the concomitant randomization effect associated with the rotation of alpha particles. The

phenomenon is similar to that observed previously in Ti-6Al-4V [9] and Zn-22%Al [6]. The observed decrease in the Kearn's factor from 0.52 to 0.44 also suggests a weakening of crystallographic texture toward a random one, which would exhibit a Kearn's factor of 0.361 measured for the normal plane of tested specimens (0,333 in a perfectly-random material).

Summary and Conclusions

1. Superplastic deformation of Ti-6Al-4V with a mean grain size of 135 nm at the extremely low temperature of 550°C was investigated. Using the optimal strain rate of 2×10^{-4} s^{-1}, a total elongation of 1000% was obtained with a strain rate sensitivity and flow stress of 0.47 and 130 MPa, respectively.
2. Microstructure observations showed extensive deformation-induced grain growth during SP deformation. The specimens did not have porosity near the fracture surface.
3. The texture weakened considerably during low-temperature SP deformation. Such weakening was mirrored in the values of the Kearn's factor which decreased from 0.52 to 0.44, the latter value approaching that of a random material (i.e., 0.361)
4. The apparent activation energy under the optimal SP conditions was found to be of 149 kJ/mole at n=2 (m=0.5) indicating that the dominant deformation mechanism was grain-boundary sliding accommodated by grain-boundary diffusion and possibly matrix-dislocation activity.

Acknowledgements

This work was supported by Grant № P725 from the Russian Ministry of Science and Education.

References

[1] H. Inagaki, Enhanced superplasticity in high strength Ti alloys, Z. Metallk. 86 (1995) 643-650.

[2] G.A. Salishchev et al., Development of Ti-6Al-4V sheet with low temperature superplastic properties, J. Mater. Proc. Technol. 116 (2001) 265-268.

[3] S.V. Zherebtsov et al., Production of submicrocrystalline structure in large-scale Ti-6Al-4V billet by warm severe deformation processing, Scripta Mater. 51 (2004) 1147-1151.

[4] Y.G. Ko et al., Low-temperature superplasticity of ultra-fine-grained Ti-6AI-4V processed by equal-channel angular pressing, Metall. Mater. Trans. 37A (2006) 381-391.

[5] A.V. Sergueeva et al., Superplastic behaviour of ultrafine-grained Ti-6Al-4V alloys, Mater. Sci. Eng. A323 (2002) 318-325.

[6] O.A. Kaibyshev: *Superplasticity of Alloys, Intermetallides and Ceramics* (Springer-Verlag, Berlin 1992).

[7] G.A. Salishchev et al., Submicrocrystalline and nanocrystalline structure formation in materials and search for outstanding superplastic properties, Mater. Sci. Forum 170-172 (1994) 121-130.

[8] Y. Mishin, C. Herzig, Diffusion in the Ti-Al system, Acta Mater. 48 (2000) 589-623.

[9] S.L. Semiatin et al., Plastic flow and microstructure evolution during low-temperature superplasticity of ultrafine Ti-6Al-4V sheet material, Metall. Mater. Trans. 41A (2010) 499-512.

[10] G.A. Sargent et al., Low-temperature coarsening and plastic flow behavior of an α/β titanium billet material with an ultrafine microstructure, Metall. Mater. Trans. 39A (2008) 2949-2964.

[11] G. Wasserman and I. Greven: *Texturen metallischer Werkstoffe* (Springer-Verlag, Berlin 1962).

[12] M.J. Donachie, Jr.: *Titamium: a Technical Guide* (ASM International, USA 2000).

[13] A. Arieli, A. Rosen, Superplastic deformation of Ti-6Al-4V, Metall. Trans. 8A (1977) 1591-1596.